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(54) FINE PARTICLES FOR SUN PROTECTION, COATING LIQUID FOR FORMING SUN PROTECTION FILM CONTAINING THE SAME AND SUN PROTECTION FILM

(57)Abstract:
PROBLEM TO BE SOLVED: To provide fine particles for sun protection and a coating liquid for forming a sun protection film capable of forming a sun protection film having a high visible light transmittance, a low solar radiation transmittance and a low haze value and to provide a sun protection film.
SOLUTION: The fine particles for sun protection are fine indium-tin oxide particles having a powder color L* of 52-80, a* of -10 to -0.1 and b* of -14 to +20 in the L*a*b* color system and having ≥55 m2/g specific surface area. The coating liquid for forming a sun protection film contains the fine indium-tin oxide particles having the above properties. The sun protection film is obtained by heat-treating the coating liquid applied on a substrate.

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* NOTICES *

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CLAIMS

[Claim(s)]
[Claim 1] The particle for solar radiation electric shielding to which L* of the fine-particles color in a L*a*b* color coordinate system is characterized by consisting of indium stannic acid ghost particles -10--0.1 and whose b* 52-80, and a* are -14-20 and, whose specific surface area is more than 55m2/g in the particle for solar radiation electric shielding.
[Claim 2] Coating liquid for solar radiation screen formation characterized by distributing the particle for solar radiation electric shielding according to claim 1 in a solvent in the coating liquid for solar radiation screen formation for forming a solar radiation screen.
[Claim 3] Coating liquid for solar radiation screen formation according to claim 2 characterized by an inorganic binder or a resin binder containing as a binder component.
[Claim 4] The solar radiation screen characterized by the above-mentioned coating liquid for solar radiation screen formation consisting of coating liquid for solar radiation screen formation according to claim 2 or 3 in the solar radiation screen which heat-treats the coating liquid for solar radiation screen formation applied on the base material, and is formed.
[Claim 5] The solar radiation screen characterized by forming silicon, a zirconium, titanium, or the oxide film of aluminum on a solar radiation screen according to claim 4.
[Claim 6] The solar radiation screen according to claim 4 or 5 characterized by for the solar radiation permeability in 300-2100nm of wavelength regions at the time of 80% or more of light permeability being less than 60%, and a haze value being less than 1%.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the coating liquid for solar-radiation screen formation and the solar-radiation screen containing the particle for solar-radiation electric shielding used for glass, such as apertures, such as a car, a building, an office, and a general residence, a telephone booth, a show window, a lamp for lighting, and a transparence case, and the transparence base material which needs another plastics and solar-radiation electric-shielding function, and this particle, and relates to the improvement of the particle for solar-radiation electric shielding which enables formation of a solar-radiation screen with the low Hayes value especially, the coating liquid for solar-radiation screen formation, and a solar-radiation screen
[0002]
[Description of the Prior Art] Forming the film which consists of an ingredient which reflects infrared radiation in a glass front face, and considering as heat reflective glass conventionally, by making a heat component into the approach of removing and decreasing, from the external light sources, such as sunlight and an electric bulb, was performed. The metallic material which has free electrons, such as metallic oxides, such as FeOx, CoOx, CrOx, and TiOx, and Ag, Au, Cu, nickel, aluminum, so much has been chosen as the ingredient.
[0003] However, with these ingredients, since the light also had the property reflected or absorbed in coincidence in addition to the infrared radiation greatly contributed to a thermal effect, there was a problem to which light permeability falls. And in the transparence base material used for building materials, a vehicle, a telephone booth, etc., since the high permeability of a light field is needed, when using the above-mentioned ingredient, thickness must be made very thin. For this reason, being formed membranes and used for the thin film of 10nm level using the physical forming-membranes methods, such as spray printing, a CVD method, or a spatter, vacuum evaporation technique, has usually been performed.
[0004] However, these membrane formation approaches need large-scale equipment and a vacuum facility, and a problem is in productivity or large area-ization, and they have the fault that a membranous manufacturing cost is high. Moreover, if it is going to make high a solar radiation electric shielding property (property which covers the light of 300-2100nm of wavelength regions) with these ingredients, the inclination which becomes high is in coincidence, an appearance [GIRAGIRA / appearance] like a mirror will be given and the reflection factor of a light field will also spoil a fine sight. Furthermore, with these ingredients, when there is much what has membranous high conductivity and membranous conductivity is high, the electric wave of a cellular phone, TV, radio, etc., etc. is reflected, it becomes non-receipt or there are problems, such as causing an electromagnetic interference in a circumference area.
[0005] In order to have improved such a conventional fault, as a membranous physical property, the reflection factor of the light of a light field was low, and the reflection factor of an infrared field was high, and membranous conductivity needed to form the more controllable film in general than [106ohms /] **.
[0006] By the way, the antimony stannic acid ghost and the indium stannic acid ghost (it abbreviates to ITO hereafter) are known as an ingredient in which light transmission has the solar radiation electric shielding function in which it moreover excelled highly. And these ingredients do not give the appearance [GIRAGIRA / the light reflection factor / appearance / comparatively low]. However, since a plasma frequency was in a near infrared ray field, reflection and the absorption effect of these film in the near-infrared region near the light were not enough.
[0007] Then, the approach which enabled it to also reflect and absorb light of the near-infrared region near the

light is proposed by JP,7-69652,A and JP,8-41441,A by using the ITO powder of a specific color heat-treated in inert gas or weak reducibility gas. And according to this approach, low solar radiation permeability is obtained, maintaining high light permeability. However, an ingredient which forms the film of the Hayes value which is less than 1% was not yet realized.

[0008] The above-mentioned Hayes value is the rate of the diffuse-transmission light to all the transmitted lights, and when this value is high, it blooms cloudy and is visible to human being's eyes. Therefore, for the aperture material as which transparency is required, especially the car application which needs transparency more, film of the Hayes value which is less than 1% was desired.

[0009]

[Problem(s) to be Solved by the Invention] The place which this invention was made paying attention to such a trouble, and is made into the technical problem It is in offering the particle for solar radiation electric shielding which enables formation of a solar radiation screen with solar radiation permeability light permeability is high and low and low extent in which the Hayes value is moreover less than 1%. Furthermore, it is in offering the coating liquid for solar radiation screen formation which can form membranes by the simple applying method, without using the physical forming-membranes method of high cost, and the solar radiation screen using this.

[0010]

[Means for Solving the Problem] Then, it came to find out the phenomenon in which discover plasma absorption strong against the near-infrared region near a light field while having the maximum of permeability in a light field by production of the film which distributed the ITO particle of high specific surface area having [and] a specific fine-particles color when this invention person etc. inquired wholeheartedly, in order to solve the above-mentioned technical problem to altitude, and the Hayes value becomes very low [having the minimum of permeability], and this invention was completed. Here, for L* of the fine-particles color in the L*a*b* color coordinate system (JIS Z8729) which Commission Internationale de l'Eclariage (CIE) recommends the specific fine-particles color, 52-80, and a* are [-10--0.1 and b*] -14-20, and the specific surface area of a particle is more than 55m2/g.

[0011] That is, 52-80, and a* are [-10--0.1 and b*] -14-20, and L* of a fine-particles color [in / on the assumption that the particle for solar radiation electric shielding / in invention concerning claim 1 / a L*a*b* color coordinate system] is characterized by specific surface area consisting of indium stannic acid ghost particles which are more than 55m2/g.

[0012] Next, invention which invention concerning claim 2 is characterized by distributing the particle for solar radiation electric shielding according to claim 1 in a solvent on the assumption that the coating liquid for solar radiation screen formation for forming a solar radiation screen, and relates to claim 3 is characterized by an inorganic binder or a resin binder containing as a binder component on the assumption that the coating liquid for solar radiation screen formation concerning invention according to claim 2.

[0013] Moreover, invention concerning claim 4 is premised on the solar radiation screen which heat-treats the coating liquid for solar radiation screen formation applied on the base material, and is formed. Invention which is characterized by the above-mentioned coating liquid for solar radiation screen formation consisting of coating liquid for solar radiation screen formation according to claim 2 or 3, and relates to claim 5 Invention which is characterized by forming silicon, a zirconium, titanium, or the oxide film of aluminum on a solar radiation screen according to claim 4, and relates to claim 6 It is characterized by for the solar radiation permeability in 300-2100nm of wavelength regions at the time of 80% or more of light permeability being less than 60%, and a haze value being less than 1% a premise [the solar radiation screen concerning invention according to claim 4 or 5].

[0014]

[Embodiment of the Invention] Hereafter, the gestalt of operation of this invention is explained to a detail.

[0015] First, in the ITO particle which constitutes the particle for solar radiation electric shielding concerning this invention, although the tin content in element conversion is principle arbitration, if it is possible, its 1 - 15 % of the weight is desirable. It is because dissolving [of tin] may become inadequate at less than 1 % of the weight when the addition effectiveness of tin may not be seen and it exceeds 15 % of the weight. Moreover, it requires that the specific surface area of the above-mentioned ITO particle is more than 55m2/g as mentioned above. the above-mentioned optical property considered as a request obtains under by 55m2/g -- having -- it is because there is nothing.

[0016] Moreover, as the fine-particles color of the ITO particle which constitutes the particle for solar radiation electric shielding concerning this invention was mentioned above, although L* of the fine-particles color in a L*a*b* color coordinate system requires that 52-80, and a* are [-10--0.1 and b*] -14-20, it explains the reason below.
[0017] If the interaction of the electron in a general light and the matter is explained, there is a plasma frequency of a proper in a certain matter, the light of long wavelength is reflected from this frequency, and it is known that the light of short wavelength will be penetrated.
[0018] Plasma frequency omegap is expressed with the following formulas (1).
[0019] omegap2=nq2/epsilonm (1)
Here, for n, a conduction electron consistency and q are [a dielectric constant and m of electronic charge and epsilon] electronic effective masses.
[0020] Since a plasma frequency will generally become large if a conduction electron consistency increases, it will be reflected to the light by the side of short wavelength. Since a visible ray is penetrated by ITO although a reflection factor is already high from a light field with a metal since conduction electron consistencies are three 1022-/cm and1021/three cm in ITO with a metal, and a near infrared ray region to a reflection factor becomes high, it can use as a solar radiation screen.
[0021] if an ITO particle is processed by the mixed gas of alcoholic content inert gas or reducibility gas, and inert gas as indicated by JP,8-41441,A -- the fine-particles color -- yellow -> yellowish green -> light blue -> dark blue -> dark blue -> -- the powder-compacting resistance also decreases at the same time it changes that it is black. This is considered that the free electron increased by the increment in a hole in order to generate a hole and to work as a donor to the indium oxide this hole of whose is a n-type semiconductor like tin by processing ITO by the above gas, and it is expected that there is close, a fine-particles color and a conduction electron consistency, i.e., a plasma frequency, relation.
[0022] Then, the place which searched for the conditions for investigating the relation between the fine-particles color of an ITO particle and specific surface area, and the solar radiation permeability when forming membranes in detail, and attaining high solar radiation electric shielding, If -10--0.1 and b* are set to -14-20 by 52-80, and a*, L* of the fine-particles color by the L*a*b* color coordinate system in the ITO particle more than specific-surface-area of 55m 2/g It was checked that solar radiation permeability becomes low with less than 60%, maintaining high light permeability, and the Hayes value moreover becomes less than 1%.
[0023] Next, although the coating liquid for solar radiation screen formation concerning this invention distributes an ITO particle in a solvent, especially a solvent is not limited, and when spreading conditions, a spreading environment, and an inorganic binder and a resin binder are made to contain, it is suitably chosen according to a binder component. For example, it is usable, and if needed, various kinds of organic solvents called ketones, such as ether, such as alcohols, such as water, ethanol, propanol, a butanol, isopropyl alcohol, isobutyl alcohol, and diacetone alcohol, methyl ether, ethyl ether, and the propyl ether, ester, an acetone, a methyl ethyl ketone, a diethyl ketone, a cyclohexanone, and an isobutyl ketone, may add an acid and alkali, and may carry out pH adjustment. Furthermore, since the distributed stability of the particle in coating liquid is raised further, of course, addition of various kinds of surfactants, a coupling agent, etc. is also possible.
[0024] Moreover, although especially the class of an inorganic binder or resin binder is not limited, as an inorganic binder, it can use a metal alkoxide, and these partial hydrolysis condensation polymerization objects or the ORGANO silazane of silicon, a zirconium, titanium, or aluminum, and can use thermosetting resin, such as thermoplastics, such as acrylic resin, and an epoxy resin, etc. as a resin binder.
[0025] Next, especially if the method of distributing an ITO particle in a solvent is the approach of distributing to homogeneity in coating liquid, it will not be limited, for example, a bead mill, a ball mill, a sand mill, a paint shaker, an ultrasonic homogenizer, etc. are mentioned.
[0026] Since conductivity of the film when forming the film using this coating liquid is performed along with the electric conduction pass which went via the contact part of an ITO particle, it is easy to be able to cut electric conduction pass partially by adjusting the amount of a surfactant or a coupling agent, and to reduce membranous conductivity to the surface-electrical-resistance value 106ohms / more than **. Moreover, conductivity is controllable also by the degree of the content of an inorganic binder or a resin binder.
[0027] Next, on a base material, the above-mentioned ITO particle which constitutes the particle for solar radiation electric shielding deposits the solar radiation screen concerning this invention on high density, it forms

the film, and the resin binder or the inorganic binder contained in coating liquid is effective in raising the adhesion to the base material of an ITO particle after spreading and hardening, and raising a membranous degree of hardness further. Moreover, the degree of hardness of binding capacity or the film and weatherability can be further raised to the base material of the film which uses an ITO particle as a principal component by putting the coat which consists of silicon, a zirconium, titanium or metal alkoxides of aluminum, and these partial hydrolysis condensation polymerization objects further as the 2nd layer, and forming silicon, a zirconium, titanium, or the oxide film of aluminum on the film obtained by doing in this way.

[0028] Moreover, the film obtained when a resin binder or an inorganic binder is not included in coating liquid becomes the membrane structure which only the above-mentioned ITO particle deposited on the base material. Although a solar radiation shielding effect is shown also as [this], it is good to apply the coating liquid which contains an inorganic binder or resin binders, such as silicon, a zirconium, titanium, or metal alkoxides of aluminum, these partial hydrolysis condensation polymerization objects, further, to form a coat on this film, and to consider as multilayers. Since a coating liquid component fills the gap which the ITO particle of the 1st layer deposited and is formed by doing in this way, membranous Hayes decreases, and light permeability improves, and the binding property to the base material of a particle improves.

[0029] As an approach of binding the film top which only the above-mentioned ITO particle deposited, or an ITO particle with the coat which consists of silicon, a zirconium, titanium, or the metal alkoxides and these partial hydrolysis condensation polymerization objects of aluminum on the film used as a principal component, the ease of a membrane formation process or the viewpoint of cost to the applying method is effective. 40 or less % of the weight of coating liquid in [all] a solution is desirable at the oxide conversion from which two or more sorts are included and the content is obtained [one sort or] after heating in water or alcohol in silicon, a zirconium, titanium, and the metal alkoxides and these partial hydrolysis condensation polymerization objects of aluminum. Moreover, it is also possible to add an acid and alkali and to carry out pH adjustment if needed. It is possible to produce oxide coats, such as silicon, a zirconium, titanium, and aluminum, easily by applying such liquid as the 2nd layer further on the film which uses an ITO particle as a principal component, and heating it. Furthermore, the ORGANO silazane solution may be used as the binder component used for the coating liquid of this invention, or coating liquid for overcoats.

[0030] Especially the method of application for coat formation of the oxide coat used by the method of application and this invention of coating liquid concerning this invention is not limited. For example, which approach may be used as long as a spin coat method, the bar coat method, a spray coating method, a dip coating method, screen printing, the roll coat method, flow coating, etc. are flatness and the approach of applying to homogeneity thinly about processing liquid.

[0031] And whenever [after spreading of the coating liquid containing the metal alkoxide and its hydrolysis polymerization object of silicon, a zirconium, titanium, or aluminum / base material stoving temperature] as an inorganic binder Since the polymerization reaction of the alkoxide contained in a paint film at less than 100 degrees C or its hydrolysis polymerization object remains by un-completing in many cases, and water and an organic solvent remain in the film and it becomes the cause of reduction of the light permeability of the film after heating 100 degrees C or more heat desirable still more preferably above the boiling point of the solvent in coating liquid.

[0032] Moreover, what is necessary is just to make it harden according to each hardening approach, when a resin binder is used. For example, what is necessary is just to leave it as it is after spreading that what is necessary is just to irradiate ultraviolet rays suitably if it is ultraviolet-rays hardening resin, if it is room-temperature-setting resin. For this reason, spreading in the site to the existing windowpane etc. is possible.

[0033] And since the ITO particle is distributing in the solar radiation screen concerning this invention, there is little reflection in a light field compared with the film in which a crystal has the mirror plane-like front face which filled the inside of the film precisely like the oxide thin film manufactured by the physical forming-membranes method, and it can avoid presenting the appearance [GIRAGIRA / appearance]. Moreover, since visible [as mentioned above] and it has a plasma frequency in a near-infrared region on the other hand, the plasma reflection accompanying this becomes large in a near-infrared region.

[0034] Moreover, multilayers of 1% or less of luminous reflectances can be easily obtained by forming film of a low refractive index like SiO2 or MgF2 on the solar radiation screen concerning this invention to control reflection of a light field further.

[0035] Moreover, in order to make the further ultraviolet-rays electric shielding function of the particle for solar radiation electric shielding concerning this invention, coating liquid, and a solar radiation screen give, one sort, such as particles, such as titanium oxide of an inorganic system, and a zinc oxide, cerium oxide, a benzophenone of an organic system, and benzotriazol, or two sorts or more may be added.
[0036] According to this invention, manufacture of the solar radiation screen which demonstrates a solar radiation shielding effect by using the ITO particle which has the property mentioned above as a particle for solar radiation electric shielding is possible, but since this ITO particle is an inorganic material, even if weatherability is very high compared with an organic material, for example, it uses it for the part to which sunrays (ultraviolet rays) hit, degradation of a color or many functions is hardly produced. Moreover, since the coating liquid for solar radiation screen formation concerning this invention is not what forms the above-mentioned solar radiation screen using the decomposition or the chemical reaction of a spreading component by the heat at the time of baking, it can form the transparency film of the uniform thickness by which the property was stabilized.
[0037]
[Example] Hereafter, the example of this invention is explained concretely. However, this invention is not limited to the following examples.
[0038] In addition, the color (standard source C, 10-degree visual field) of the light permeability and solar radiation permeability of the obtained solar radiation screen, and fine particles was measured using the spectrophotometer U-4000 by Hitachi, Ltd. moreover, a haze value -- the Murakami Color Research Laboratory make -- it measured using HR-200. The light permeability of three kinds of film with which membranes are formed by three sorts of bar coating machines from which a wire size differs in film evaluation, and the obtained thickness differs, solar radiation permeability, and a haze value were measured, respectively, and the solar radiation permeability and haze value at the time of 84% of light permeability were calculated from the three-point plot.
[0039] The various ITO particles which have a fine-particles color as shown in the following table 1 for selection of the ITO particle applied as a particle for selection solar radiation electric shielding of ITO, and specific surface area were prepared.
[0040] In addition, in Table 1, it is the ITO particle which requires a-e for an example, and is the ITO particle which requires f-l for the example of a comparison.
[0041]
[Table 1]

	粉体色			比表面積
ITO	L*	a*	b*	(m^2/g)
a	66.228	-5.865	-3.495	79.3
b	62.460	-5.855	-3.533	73.6
c	59.308	-5.418	0.867	77.6
d	59.452	-5.591	-3.701	58.1
e	71.405	-7.608	-3.087	61.7
f	61.338	-5.264	-12.566	34.3
g	56.502	-4.733	-8.949	41.8
h	54.634	-5.240	-11.234	23.7
i	67.272	1.628	15.263	85.4
j	85.144	-4.408	25.409	29.0
k	35.597	-0.893	-4.586	26.7
l	46.372	-3.7528	-9.1242	22.4

Next, the result of having evaluated the optical property about the solar radiation screen concerning the example and the example of a comparison which were formed using the above-mentioned ITO particle is described.
[0042] The paint shaker into which 0.3mm zirconia beads equivalent to 63% of filling factors were put

distributed 20 % of the weight of ITO particles of a of the [example 1] table 1, 63.3 % of the weight of methyl isobutyl ketone, and 16.7 % of the weight of dispersants for 12 hours.
[0043] Next, after applying the coating liquid which consists of 67.5 % of the weight of obtained dispersion liquid, 27.5 % of the weight of acrylic resin solutions which dissolved in methyl isobutyl ketone as binders, and 5 % of the weight of curing agents to a 100mmx100mmx3mm soda lime glass substrate with the bar of the yarn counts 40, 24, and 6, respectively, it calcinated at 180 degrees C for 1 hour, and the solar radiation screen A was obtained. As shown in Table 2, the solar radiation permeability and haze values of the solar radiation screen A were 58% and 0.3%, respectively.
[0044] [An example 2 - an example 5 and the example 1 of a comparison - example 7 of a comparison] except having used similarly the ITO particle of b-l shown in Table 1 It is made to be the same as that of an example 1., respectively The solar radiation screen B (example 2), the solar radiation screen C (example 3) The solar radiation screen D (example 4), the solar radiation screen E (example 5), the solar radiation screen F (example 1 of a comparison) The solar radiation screen G (example 2 of a comparison), the solar radiation screen H (example 3 of a comparison), the solar radiation screen I (example 4 of a comparison), the solar radiation screen J (example 5 of a comparison), the solar radiation screen K (example 6 of a comparison), and the solar radiation screen L (example 7 of a comparison) were obtained.
[0045] And as shown in the following table 2, the solar radiation permeability of the example 1 - the example 5 was all less than 60%, and the Hayes value was less than 1%. On the other hand, all, solar radiation permeability exceeds 60% and the haze value of the example 4 of a comparison of the example 1 of a comparison - the example 7 of a comparison was 11.
[0046]
[Table 2]

		可視光透過率８４%固定	
		日射透過率(%)	ヘイズ値(%)
実施例１	膜A	58.0	0.3
実施例２	膜B	58.3	0.2
実施例３	膜C	59.0	0.2
実施例４	膜D	59.0	0.3
実施例５	膜E	58.6	0.3
比較例１	膜F	62.2	0.6
比較例２	膜G	62.0	0.7
比較例３	膜H	62.8	0.5
比較例４	膜I	69.8	11
比較例５	膜J	68.3	0.8
比較例６	膜K	63.0	0.4
比較例７	膜L	62.0	0.5

[0047]
[Effect of the Invention] For L* of a fine-particles color [in / in the particle for solar radiation electric shielding concerning this invention / a L*a*b* color coordinate system], 52-80, and a* are [-10--0.1 and b*] -14-20. And since specific surface area consists of indium stannic acid ghost particles which are more than 55m2/g, it has the effectiveness which enables formation of a solar radiation screen with solar radiation permeability light permeability is high and low and low extent in which the Hayes value is moreover less than 1%.
[0048] Moreover, it has the effectiveness that the coating liquid for solar radiation screen formation which can form membranes by the simple applying method, without using the physical forming-membranes method of high cost can be offered, and the above-mentioned solar radiation screen using this coating liquid for solar radiation screen formation can be offered, by making this particle for solar radiation electric shielding contain.

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最終頁に続く

(54)【発明の名称】　日射遮蔽用微粒子とこの微粒子を含む日射遮蔽膜形成用塗布液および日射遮蔽膜

(57)【要約】
【課題】　可視光透過率が高くて日射透過率が低く、しかもヘイズ値が低い日射遮蔽膜の形成を可能とする日射遮蔽用微粒子と日射遮蔽膜形成用塗布液および日射遮蔽膜を提供する。
【解決手段】　この日射遮蔽用微粒子は、Ｌ*ａ*ｂ*表色系における粉体色のＬ*が５２～８０、ａ*が－１０～－０．１、ｂ*が－１４～２０であり、かつ、比表面積が５５m^2／ｇ以上であるインジウム錫酸化物微粒子で構成されることを特徴とする。また、日射遮蔽膜形成用塗布液は上記特性を有するインジウム錫酸化物微粒子が含有されることを特徴とし、日射遮蔽膜は基材上に塗布された上記日射遮蔽膜形成用塗布液を熱処理して得られることを特徴とする。

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【特許請求の範囲】

【請求項１】日射遮蔽用微粒子において、

Ｌ*ａ*ｂ*表色系における粉体色のＬ*が５２～８０、ａ*が－１０～－０．１、ｂ*が－１４～２０であり、かつ、比表面積が５５m^2／ｇ以上であるインジウム錫酸化物微粒子で構成されることを特徴とする日射遮蔽用微粒子。

【請求項２】日射遮蔽膜を形成するための日射遮蔽膜形成用塗布液において、

請求項１記載の日射遮蔽用微粒子が溶媒中に分散されていることを特徴とする日射遮蔽膜形成用塗布液。

【請求項３】バインダー成分として、無機バインダーまたは樹脂バインダーが含有されることを特徴とする請求項２記載の日射遮蔽膜形成用塗布液。

【請求項４】基材上に塗布された日射遮蔽膜形成用塗布液を熱処理して形成される日射遮蔽膜において、

上記日射遮蔽膜形成用塗布液が請求項２または３記載の日射遮蔽膜形成用塗布液で構成されていることを特徴とする日射遮蔽膜。

【請求項５】請求項４記載の日射遮蔽膜上に、珪素、ジルコニウム、チタン若しくはアルミニウムの酸化物膜が形成されていることを特徴とする日射遮蔽膜。

【請求項６】可視光透過率８０％以上のときの波長域３００～２１００ｎｍにおける日射透過率が６０％未満で、かつ、ヘーズ値が１％未満であることを特徴とする請求項４または５記載の日射遮蔽膜。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、車両、ビル、事務所、一般住宅などの窓、電話ボックス、ショーウィンドー、照明用ランプ、透明ケースなど、ガラス、プラスチックスその他日射遮蔽機能を必要とする透明基材に用いる日射遮蔽用微粒子とこの微粒子を含む日射遮蔽膜形成用塗布液および日射遮蔽膜に係り、特に、ヘイズ値の低い日射遮蔽膜の形成を可能とする日射遮蔽用微粒子と日射遮蔽膜形成用塗布液および日射遮蔽膜の改善に関するものである。

【０００２】

【従来の技術】太陽光や電球などの外部光源から熱成分を除去・減少する方法として、従来、ガラス表面に赤外線を反射する材料からなる膜を形成して熱線反射ガラスとすることが行われていた。その材料にはＦｅＯｘ、ＣｏＯｘ、ＣｒＯｘ、ＴｉＯｘなどの金属酸化物やＡｇ、Ａｕ、Ｃｕ、Ｎｉ、Ａｌなどの自由電子を多量にもつ金属材料が選択されてきた。

【０００３】しかし、これらの材料では熱効果に大きく寄与する赤外線以外に、可視光も同時に反射若しくは吸収する性質があるため可視光透過率が低下する問題があった。そして、建材、乗り物、電話ボックスなどに用いられる透明基材では可視光領域の高い透過率が必要とされることから、上記材料を利用する場合は膜厚を非常に薄くしなければならない。このため、スプレー焼付けやＣＶＤ法、あるいはスパッタ法や真空蒸着法などの物理成膜法を用いて１０ｎｍレベルの薄膜に成膜して用いられることが通常行われてきた。

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【０００４】しかしながら、これらの成膜方法は大がかりな装置や真空設備を必要とし、生産性や大面積化に問題があり、膜の製造コストが高いといった欠点がある。また、これらの材料で日射遮蔽特性（波長域３００～２１００ｎｍの光を遮蔽する特性）を高くしようとすると、可視光領域の反射率も同時に高くなってしまう傾向があり、鏡のようなギラギラした外観を与えて美観を損ねてしまう。更に、これらの材料では膜の導電性が高いものが多く、膜の導電性が高いと携帯電話やＴＶ、ラジオなどの電波を反射して受信不能になったり、周辺地域に電波障害を引き起こすなどの問題がある。

【０００５】このような従来の欠点を改善するには、膜の物理特性として、可視光領域の光の反射率が低くて赤外線領域の反射率が高く、かつ膜の導電性が概ね10^6Ω/□以上に制御可能な膜を形成する必要があった。

【０００６】ところで、可視光透過率が高くしかも優れた日射遮蔽機能を持つ材料として、アンチモン錫酸化物やインジウム錫酸化物（以下、ＩＴＯと略す）が知られている。そして、これらの材料は可視光反射率が比較的低くギラギラした外観を与えることはない。但し、プラズマ周波数が近赤外線領域にあるために可視光に近い近赤外域におけるこれらの膜の反射・吸収効果が十分でなかった。

【０００７】そこで、不活性ガスあるいは弱還元性ガス中で熱処理した特定の色のＩＴＯ粉末を用いることにより、可視光に近い近赤外域の光も反射・吸収できるようにした方法が、特開平７－６９６３２号公報、特開平８－４１４４１号に提案されている。そして、この方法によれば、高い可視光透過率を維持しつつ、低い日射透過率が得られている。しかし、１％を下回るようなヘイズ値の膜を形成するような材料は未だ実現されていなかった。

【０００８】上記ヘイズ値は、全透過光に対する拡散透過光の割合であり、この値が高いと人間の目には曇って見える。したがって、透明性が要求される窓材、特により透明性を必要とする車両用途では、１％を下回るヘイズ値の膜が望まれていた。

【０００９】

【発明が解決しようとする課題】本発明はこの様な問題点に着目してなされたもので、その課題とするところは、可視光透過率が高くて日射透過率が低く、しかもヘイズ値が１％を下回る程度の低い日射遮蔽膜の形成を可能とする日射遮蔽用微粒子を提供することにあり、更には高コストの物理成膜法を用いずに簡便な塗布法で成膜できる日射遮蔽膜形成用塗布液と、これを用いた日射遮

蔽膜を提供することにある。

【００１０】

【課題を解決するための手段】そこで、上記課題を解決するため本発明者等が鋭意研究を行ったところ、特定の粉体色を有し、かつ高比表面積のＩＴＯ微粒子を高度に分散した膜の作製によって可視光領域に透過率の極大を持つと共に、可視光領域に近い近赤外域に強いプラズマ吸収を発現して透過率の極小を持ち、かつ、ヘイズ値が極めて低くなるという現象を見出すに至り本発明を完成した。ここで、特定の粉体色は、国際照明委員会（ＣＩＥ）が推奨しているＬ*ａ*ｂ*表色系（ＪＩＳ　Ｚ８７２９）における粉体色のＬ*が５２～８０、ａ*が－１０～－０．１、ｂ*が－１４～２０であり、また、微粒子の比表面積は５５m^2／ｇ以上である。

【００１１】すなわち、請求項１に係る発明は、日射遮蔽用微粒子を前提とし、Ｌ*ａ*ｂ*表色系における粉体色のＬ*が５２～８０、ａ*が－１０～－０．１、ｂ*が－１４～２０であり、かつ、比表面積が５５m^2／ｇ以上であるインジウム錫酸化物微粒子で構成されることを特徴とする。

【００１２】次に、請求項２に係る発明は、日射遮蔽膜を形成するための日射遮蔽膜形成用塗布液を前提とし、請求項１記載の日射遮蔽用微粒子が溶媒中に分散されていることを特徴とし、請求項３に係る発明は、請求項２記載の発明に係る日射遮蔽膜形成用塗布液を前提とし、バインダー成分として、無機バインダーまたは樹脂バインダーが含有されることを特徴とするものである。

【００１３】また、請求項４に係る発明は、基材上に塗布された日射遮蔽膜形成用塗布液を熱処理して形成される日射遮蔽膜を前提とし、上記日射遮蔽膜形成用塗布液が請求項２または３記載の日射遮蔽膜形成用塗布液で構成されていることを特徴とし、請求項５に係る発明は、請求項４記載の日射遮蔽膜上に、珪素、ジルコニウム、チタン若しくはアルミニウムの酸化物膜が形成されていることを特徴とし、請求項６に係る発明は、請求項４または５記載の発明に係る日射遮蔽膜を前提とし、可視光透過率８０％以上のときの波長域３００～２１００ｎｍにおける日射透過率が６０％未満で、かつ、ヘーズ値が１％未満であることを特徴とする。

【００１４】

【発明の実施の形態】以下、本発明の実施の形態について詳細に説明する。

【００１５】まず、本発明に係る日射遮蔽用微粒子を構成するＩＴＯ微粒子において、元素換算での錫含有量は、原則任意であるが可能なら１～１５重量％が好ましい。１重量％未満では錫の添加効果が見られないことがあり、１５重量％を超えると錫の固溶が不十分となることがあるからである。また、上記ＩＴＯ微粒子の比表面積は上述したように５５m^2／ｇ以上であることを要する。５５m^2／ｇ未満では所望とする上記光学特性が得られないからである。

【００１６】また、本発明に係る日射遮蔽用微粒子を構成するＩＴＯ微粒子の粉体色は、上述したようにＬ*ａ*ｂ*表色系における粉体色のＬ*が５２～８０、ａ*が－１０～－０．１、ｂ*が－１４～２０であることを要するが、その理由は以下に説明する。

【００１７】一般的な光と物質内の電子の相互作用について説明すると、ある物質には固有のプラズマ周波数があってこの周波数より長波長の光は反射され、短波長の光は透過されることが知られている。

【００１８】プラズマ周波数ω_pは以下の式（１）で表される。

【００１９】$\omega_p^2 = nq^2/\varepsilon m$　　（１）

ここで、ｎは伝導電子密度、ｑは電子の電荷、εは誘電率、ｍは電子の有効質量である。

【００２０】一般に、伝導電子密度が増加するとプラズマ周波数が大きくなるため、短波長側の光まで反射されることになる。伝導電子密度は金属で10^{22}／cm^3台、ＩＴＯで10^{21}／cm^3台であるため、金属では可視光領域からすでに反射率が高いが、ＩＴＯでは、可視光線は透過し近赤外線域から反射率が高くなるため、日射遮蔽膜として用いることができる。

【００２１】特開平８－４１４４１号にも記載されているように、ＩＴＯ微粒子をアルコール含有不活性ガス、若しくは還元性ガスと不活性ガスとの混合ガスで処理すると、その粉体色が黄色→黄緑色→淡青色→濃青色→暗青色→黒色と変化すると同時に、その圧粉抵抗も減少する。これは、ＩＴＯを前記のようなガスで処理することによって空孔が生じ、この空孔が錫と同様にｎ型半導体である酸化インジウムに対してドナーとして働くために空孔の増加によって自由電子が増加したと考えられ、粉体色と伝導電子密度、つまりプラズマ周波数とは深い関係があることが予想されている。

【００２２】そこで、ＩＴＯ微粒子の粉体色および比表面積と、成膜したときの日射透過率との関係を詳しく調査して高日射遮蔽を達成するための条件を求めたところ、比表面積５５m^2／ｇ以上のＩＴＯ微粒子におけるＬ*ａ*ｂ*表色系による粉体色のＬ*が５２～８０、ａ*が－１０～－０．１、ｂ*が－１４～２０となると、高い可視光透過率を維持しつつ日射透過率が６０％未満と低くなり、しかもヘイズ値が１％未満となることが確認された。

【００２３】次に、本発明に係る日射遮蔽膜形成用塗布液は、ＩＴＯ微粒子を溶媒中に分散したものであるが、溶媒は特に限定されるものではなく、塗布条件、塗布環境、および無機バインダーや樹脂バインダーを含有させたときはバインダー成分に合わせて適宜選択する。例えば、水やエタノール、プロパノール、ブタノール、イソプロピルアルコール、イソブチルアルコール、ジアセトンアルコールなどのアルコール類、メチルエーテル，エ

チルエーテル,プロピルエーテルなどのエーテル類、エステル類、アセトン、メチルエチルケトン、ジエチルケトン、シクロヘキサノン、イソブチルケトンなどのケトン類といった各種の有機溶媒が使用可能であり、また必要に応じて酸やアルカリを添加してｐＨ調整してもよい。さらに、塗布液中の微粒子の分散安定性を一層向上させるため、各種の界面活性剤、カップリング剤などの添加も勿論可能である。

【００２４】また、無機バインダーや樹脂バインダーの種類は特に限定されるものではないが、無機バインダーとしては、珪素、ジルコニウム、チタン、若しくはアルミニウムの金属アルコキシドやこれらの部分加水分解縮重合物あるいはオルガノシラザンが利用でき、樹脂バインダーとしてはアクリル樹脂などの熱可塑性樹脂、エポキシ樹脂などの熱硬化性樹脂などが利用できる。

【００２５】次に、ＩＴＯ微粒子を溶媒中に分散させる方法は、塗布液中に均一に分散する方法であれば特に限定されず、例えばビーズミル、ボールミル、サンドミル、ペイントシェーカー、超音波ホモジナイザーなどが挙げられる。

【００２６】この塗布液を用いて膜を形成したときの膜の導電性は、ＩＴＯ微粒子の接触個所を経由した導電パスに沿って行われるため、例えば、界面活性剤やカップリング剤の量を加減することで導電パスを部分的に切断することができ、膜の導電性を10^6Ω/□以上の表面抵抗値へ低下させることは容易である。また、無機バインダーあるいは樹脂バインダーの含有量の加減によっても導電性を制御できる。

【００２７】次に、本発明に係る日射遮蔽膜は、基材上に日射遮蔽用微粒子を構成する上記ＩＴＯ微粒子が高密度に堆積して膜を形成するものであり、塗布液中に含まれる樹脂バインダーまたは無機バインダーは、塗布、硬化後にＩＴＯ微粒子の基材への密着性を向上させ、さらに膜の硬度を向上させる効果がある。また、このようにして得られた膜上に、さらに珪素、ジルコニウム、チタン、若しくはアルミニウムの金属アルコキシド、これらの部分加水分解縮重合物からなる被膜を第２層として被着し、珪素、ジルコニウム、チタン、若しくはアルミニウムの酸化物膜を形成することで、ＩＴＯ微粒子を主成分とする膜の基材へ結着力や膜の硬度、耐候性を一層向上させることができる。

【００２８】また、塗布液中に樹脂バインダーまたは無機バインダーを含まない場合に得られる膜は、基材上に上記ＩＴＯ微粒子のみが堆積した膜構造になる。このままでも日射遮蔽効果を示すが、この膜上にさらに珪素、ジルコニウム、チタン、若しくはアルミニウムの金属アルコキシドやこれらの部分加水分解縮重合物などの無機バインダーまたは樹脂バインダーを含む塗布液を塗布して被膜を形成して多層膜とするとよい。このようにすることにより、塗布液成分が第１層のＩＴＯ微粒子の堆積した間隙を埋めて成膜されるため、膜のヘイズが低減して可視光透過率が向上し、また微粒子の基材への結着性が向上する。

【００２９】上記ＩＴＯ微粒子のみが堆積した膜上あるいはＩＴＯ微粒子を主成分とする膜上に、珪素、ジルコニウム、チタン、若しくはアルミニウムの金属アルコキシドやこれらの部分加水分解縮重合物からなる被膜で結着する方法としては、成膜工程の容易さやコストの観点から塗布法が有効である。塗布液は、水やアルコール中に珪素、ジルコニウム、チタン、アルミニウムの金属アルコキシドやこれらの部分加水分解縮重合物を１種若しくは２種以上含むものであり、その含有量は加熱後に得られる酸化物換算で全溶液中の４０重量％以下が好ましい。また、必要に応じて酸やアルカリを添加してｐＨ調整することも可能である。このような液をＩＴＯ微粒子を主成分とする膜上にさらに第２層として塗布し加熱することで、珪素、ジルコニウム、チタン、アルミニウムなどの酸化物被膜を容易に作製することが可能である。さらには、本発明の塗布液に使用するバインダー成分として、あるいはオーバーコート用の塗布液として、オルガノシラザン溶液を用いてもよい。

【００３０】本発明に係る塗布液の塗布方法および本発明で用いる酸化物被膜などの被膜形成用の塗布方法は特に限定されない。例えば、スピンコート法、バーコート法、スプレーコート法、ディップコート法、スクリーン印刷法、ロールコート法、流し塗りなど、処理液を平坦かつ薄く均一に塗布できる方法であればいずれの方法でもよい。

【００３１】そして、無機バインダーとして、珪素、ジルコニウム、チタン、若しくはアルミニウムの金属アルコキシドおよびその加水分解重合物を含む塗布液の塗布後における基材加熱温度は、１００℃未満では塗膜中に含まれるアルコキシドまたはその加水分解重合物の重合反応が未完結で残る場合が多く、また水や有機溶媒が膜中に残留して加熱後の膜の可視光透過率の低減の原因となるので、１００℃以上が好ましく、さらに好ましくは塗布液中の溶媒の沸点以上で加熱を行う。

【００３２】また、樹脂バインダーを使用した場合は、それぞれの硬化方法に従って硬化させればよい。例えば、紫外線硬化樹脂であれば紫外線を適宜照射すればよく、また常温硬化樹脂であれば塗布後そのまま放置しておけばよい。このため、既存の窓ガラスなどへの現場での塗布が可能である。

【００３３】そして、本発明に係る日射遮蔽膜ではＩＴＯ微粒子が分散しているため、物理成膜法により製造された酸化物薄膜のように結晶が緻密に膜内を埋めた鏡面状表面をもつ膜に比べると可視光領域での反射が少なく、ギラギラした外観を呈することが回避できる。また、その一方で、上記のように可視から近赤外域にプラズマ周波数をもつため、これに伴うプラズマ反射が近赤

外域で大きくなる。

【００３４】また、可視光領域の反射をさらに抑制したい場合には、本発明に係る日射遮蔽膜上に、SiO_2やMgF_2のような低屈折率の膜を成膜することにより容易に視感反射率１％以下の多層膜を得ることができる。

【００３５】また、本発明に係る日射遮蔽用微粒子、塗布液、日射遮蔽膜のさらなる紫外線遮蔽機能を付与させるため、無機系の酸化チタンや酸化亜鉛、酸化セリウムなどの微粒子や、有機系のベンゾフェノンやベンゾトリアゾールなどの１種若しくは２種以上を添加してもよい。

【００３６】本発明によれば、上述した特性を有するＩＴＯ微粒子を日射遮蔽用微粒子として用いることによって日射遮蔽効果を発揮する日射遮蔽膜の製造が可能であるが、このＩＴＯ微粒子は無機材料であるので有機材料と比べて耐候性は非常に高く、例えば太陽光線（紫外線）の当たる部位に使用しても色や諸機能の劣化はほとんど生じない。また、本発明に係る日射遮蔽膜形成用塗布液は、焼成時の熱による塗布成分の分解あるいは化学反応を利用して上記日射遮蔽膜を形成するものではないため、特性の安定した均一な膜厚の透過膜を形成することができる。

【００３７】

【実施例】以下、本発明の実施例について具体的に説明する。但し、本発明は以下の実施例に限定されるものでない。

【００３８】尚、得られた日射遮蔽膜の可視光透過率や日射透過率および粉体の色彩（標準光源Ｃ，１０°視野）は日立製作所（株）製の分光光度計Ｕ－４０００を用いて測定した。また、ヘーズ値は村上色彩技術研究所（株）製ＨＲ－２００を用いて測定した。膜評価においては線径の異なる３種のバーコーターで成膜し、得られた膜厚が異なる３種類の膜の可視光透過率、日射透過率、ヘーズ値をそれぞれ測定し、可視光透過率８４％のときの日射透過率およびヘーズ値を３点プロットから求めた。

【００３９】ＩＴＯの選定

日射遮蔽用微粒子として適用するＩＴＯ粒子の選定のために、以下の表１に示すような粉体色と比表面積を有する種々のＩＴＯ粒子を準備した。

【００４０】尚、表１において、ａ～ｅが実施例に係るＩＴＯ微粒子であり、ｆ～ｌが比較例に係るＩＴＯ微粒子である。

【００４１】

【表１】

	粉体色			比表面積
ITO	L*	a*	b*	(m^2/g)
a	66.228	-5.865	-3.495	79.3
b	62.450	-5.855	-3.533	73.6
c	59.308	-5.418	0.867	77.6
d	59.452	-5.591	-3.701	58.1
e	71.405	-7.608	-3.087	61.7
f	61.338	-5.264	-12.566	34.3
g	56.502	-4.733	-8.949	41.8
h	54.634	-5.240	-11.234	23.7
i	67.272	1.628	15.263	85.4
j	85.144	-4.408	25.409	29.0
k	35.597	-0.893	-4.586	26.7
l	46.372	-3.7528	-9.1242	22.4

次に、上記ＩＴＯ微粒子を用いて形成した実施例ならびに比較例に係る日射遮蔽膜について光学特性を評価した結果について述べる。

【００４２】［実施例１］表１のａのＩＴＯ微粒子２０重量％、メチルイソブチルケトン６３．３重量％、分散剤１６．７重量％を、充填率６３％相当の０．３mmジルコニアビーズを入れたペイントシェーカーで１２時間分散した。

【００４３】次に、得られた分散液６７．５重量％、バインダーとしてメチルイソブチルケトンに溶解したアクリル樹脂溶液２７．５重量％および硬化剤５重量％から成る塗布液を、番手４０，２４，６のバーでそれぞれ１００mm×１００mm×３mmのソーダライムガラス基板に塗布した後、１８０℃で１時間焼成して日射遮蔽膜Ａを得た。表２に示すように、日射遮蔽膜Ａの日射透過率およびヘーズ値はそれぞれ５８％、０．３％であった。

【００４４】［実施例２～実施例５および比較例１～比較例７］同様に、表１に示すｂ～ｌのＩＴＯ微粒子を用いた以外は、実施例１と同様にして、それぞれ日射遮蔽膜Ｂ（実施例２）、日射遮蔽膜Ｃ（実施例３）、日射遮蔽膜Ｄ（実施例４）、日射遮蔽膜Ｅ（実施例５）、日射

遮蔽膜Ｆ（比較例１）、日射遮蔽膜Ｇ（比較例２）、日射遮蔽膜Ｈ（比較例３）、日射遮蔽膜Ｉ（比較例４）、日射遮蔽膜Ｊ（比較例５）、日射遮蔽膜Ｋ（比較例６）、および、日射遮蔽膜Ｌ（比較例７）を得た。

【００４５】そして、以下の表２に示すように、実施例１～実施例５はいずれも日射透過率が６０％未満で、かつヘイズ値は１％未満であった。一方、比較例１～比較例７はいずれも日射透過率が６０％を越えるものであり、かつ、比較例４のヘーズ値は１１であった。

【００４６】

【表２】

		可視光透過率８４%固定	
		日射透過率(%)	ヘイズ値(%)
実施例１	膜Ａ	58.0	0.3
実施例２	膜Ｂ	58.3	0.2
実施例３	膜Ｃ	59.0	0.2
実施例４	膜Ｄ	59.0	0.3
実施例５	膜Ｅ	58.6	0.3
比較例１	膜Ｆ	62.2	0.6
比較例２	膜Ｇ	62.0	0.7
比較例３	膜Ｈ	62.8	0.5
比較例４	膜Ｉ	69.8	11
比較例５	膜Ｊ	68.3	0.8
比較例６	膜Ｋ	63.0	0.4
比較例７	膜Ｌ	62.0	0.5

【００４７】

【発明の効果】本発明に係る日射遮蔽用微粒子は、Ｌ*ａ*ｂ*表色系における粉体色のＬ*が５２～８０、ａ*が－１０～－０．１、ｂ*が－１４～２０であり、かつ、比表面積が５５m²／ｇ以上であるインジウム錫酸化物微粒子で構成されるため、可視光透過率が高くて日射透過率が低く、しかもヘイズ値が１％を下回る程度の低い日射遮蔽膜の形成を可能とする効果を有する。

【００４８】また、この日射遮蔽用微粒子を含有させることにより高コストの物理成膜法を用いずに簡便な塗布法で成膜できる日射遮蔽膜形成用塗布液を提供でき、かつ、この日射遮蔽膜形成用塗布液を用いた上記日射遮蔽膜を提供できる効果を有する。

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